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NATIONAL BUREAU OF STANDARDS REPORT

10 012

Progress Report

August 1, 1968 through January 31, 1969

THE ADHERENCE OF PORCELAIN ENAMEL TO ALUMINUM

NBS

U.S. DEPARTMENT OF COMMERCE

NATIONAL BUREAU OF STANDARDS

THE NATIONAL BUREAU OF STANDARDS

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NATIONAL BUREAU OF STANDARDS REPORT

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Progress Report

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THE ADHERENCE OF PORCELAIN ENAMEL TO ALUMINUM

by

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National Bureau of Standards

U.S. DEPARTMENT OF COMMERCE

NATIONAL BUREAU OF STANDARDS

I. Introduction

This report covers the 6 month period of August 1968 through January 1969. During this time, the Research Associateship Program of the Porcelain Enameled Aluminum Council of the Porcelain Enamel Institute has continued its endeavor to understand the mechanisms of adherence of porcelain enamel to aluminum. The resistance to spalling, which is the flaking or chipping off of the porcelain enamel from the aluminum, particularly after exposure to aggressive environments, is the criteria most often used to judge adherence. In the past six months the nature of spalling has become more closely understood. Thus, we are continuing to make progress toward the ultimate goal; to solve or prevent the problem of spalling or to be able to accurately predict its occurrence by testing prior to the placement of enameled aluminum into service.

In the summary which follows, the basic findings of the last six months are presented along with the theories of adherence developed to date. The remaining parts of the report discuss methods and future work. Two NBS reports, numbered 9533 and 9901, precede this report. They cover the results of the program from its beginning in August of 1966 to the date of this report and should be used for additional reference.

II. Summary

Much of the work done with the electron microprobe, electron microscope, etc. tended to confirm and strengthen the conclusion that magnesium was concentrating at the interface area and in some manner causing spalling. Examination of a spalled sample by microprobe showed that magnesium was present both on the interface side of the spalled enamel flake and on the surface of the bared metal. Assuming a magnesium or magnesium rich layer was present at the interface, this would indicate that spall failure occurred in this layer or area.

Using this information, and information gained previously, theories of adhesion and the cause of spalling have been developed:

1. If the alloy being enameled is commercially pure aluminum or a magnesium free alloy the oxide layer on the aluminum will be essentially Al_2O_3. During the firing process, the molten enamel will either dissolve or partially dissolve this layer and take it into solution. In either case, adherence and resistance to spall will be good. If the enamel dissolves essentially all of the layer, an equilibrium condition is set up whereby the Al_2O_3 is taken into solution by the porcelain enamel as fast as additional aluminum oxide is formed on the surface of the aluminum by the action of the molten glass. This would be the ideal condition to achieve. If, however, only partial solution is achieved, the enamel still will adhere tightly

to the Al_2O_3 layer which in turn is very adherent to its base metal. This condition should also yield good spall resistance as the undissolved Al_2O_3 is fairly resistant to corrosive attack. It might, however, tend to be weaker under impact than a completely dissolved or equilibrium condition as described above.

2. If the alloy being enameled contains magnesium, adherence problems and spalling may result. Magnesium migrates from the alloy to the interface between the alloy and the porcelain enamel during the firing of the enamel. The migration occurs faster than the enamel can take it into solution. The result is a magnesium enriched layer at the interface after firing has been completed. This layer is not adherent to the underlying aluminum oxide or aluminum alloy and is also quite susceptible to corrosive attack by moisture and various salts contained in the atmosphere. The corrosion of this layer with the attendant evolution of gases and corrosion products or the hydration of the layer due to atmospheric moisture create sufficient internal pressure to force the enamel away from the metal. Heat may also be a contributing factor by causing thermal expansion of hydrates previously formed.

3. The addition of chromium or chromates to the surface of the alloy prior to porcelain enameling helps somewhat when magnesium bearing alloys are being enameled. The chromium may act as a partial barrier to magnesium migration or may react with the magnesium present to form compounds more readily soluble by the porcelain enamel or less easily corroded by moisture, etc. after firing. Chromium may also act as an effective barrier to Mg migration by being oxidized itself instead of allowing Mg to oxidize. This would be effective if Mg does not migrate to the interface unless it is being oxidized.

III. Procedures and Discussion

A. Electron Microscopy

Previous efforts at examining the interface between porcelain enamel and aluminum had utilized 5 degree taper sections with the porcelain enamel being on the underside of the taper wedge. The taper wedge was thus aluminum, and it was felt that this thin section was being etched back away from the interface too rapidly by the various etching solutions used. This made it difficult in some cases to interpret results. Therefore, another set of samples was examined with the aluminum being on the underside and the porcelain enamel forming the wedge. (These are referred to as 175° taper sections.)

1100 prefired and enameled (excellent spall resistance), 5154 prefired and enameled (extremely poor spall resistance), 6061 prefired and enameled (poor spall resistance) and 6061 pickled and enameled (excellent spall resistance) were examined in this way, with the

the polished cross sections being given various etches before replicating in order to help interpret results.

This method gave disappointing results. The thin wedge of porcelain enamel was easily chipped and broken away from the interface, particularly during the removal of the plastic tape replicas. It also was easily removed completely by etching due to its thinness. Fig. #1 illustrates this difficulty. The sample is prefired and enameled 6061, and the polished cross section was etched with both a 4:1 dilution of Keller's Etch and a 5% NaOH solution. The porcelain enamel is the lighter material in the upper right and upper left corners. In the very bottom left portion of the micrograph the etched aluminum surface is visible. The area between the porcelain enamel and the etched aluminum surface is where the chippage or etch removal of the thin wedge has occurred, baring the essentially unetched, underlying metal. The relief at the edge of the enamel can be readily seen in this stereo view. This type of problem was encountered in most cases, although a few micrographs were free of this complicating factor. Fig. #2 is an example of a micrograph in which the enamel has not been removed. It also is of prefired and enameled 6061. The etched aluminum is in the lower part of the micrograph, and the porcelain enamel is in the upper part. The etch procedure was 5% NaOH for 15 seconds at room temperature, followed by 1% $SbCl_3$ for 30 seconds. On samples which spall, such as this one, we often see areas at the interface, such as the white areas seen here along the interface at the left.

Another method used in electron microscopy was to anodize the polished cross sections prior to replicating. This was done on 175° taper sections, with an anodizing bath of 16% H_2SO_4, 70°F and 15 volts. The anodizing time was five and ten minutes, with ten minutes appearing better. However, chippage of the thin porcelain enamel wedge was again encountered in the replicating procedure. Only preheated and enameled 1100 (excellent spall resistance) and 5154 prefired and enameled (extremely poor spall resistance) were examined. Fig. #3 is of 1100, anodized for ten minutes. The original interface runs diagonally from the lower left corner to the upper right corner. Below and to the right of this line is the aluminum surface exposed to the anodizing bath. The smooth area on the other side of the line represents unanodized aluminum which has been exposed by the removal of the thin enamel wedge in the replica stripping step. Fig. #4 is another micrograph of 1100 with the same treatment. Again the porcelain enamel has been stripped back from the original interface.

Figs. #5 and #6 are of 5154 after the same anodizing and sample preparation techniques. Again the porcelain enamel has been stripped back from the original interface, making interpretation somewhat more difficult. Note again, however, that samples such as this one of 5154 show a different type of interface than samples which do not spall.

B. Scanning Electron Microscopy

We were afforded the opportunity of examining several of our specimens with the Scanning Electron Microscope. This was done with the cooperation of Dr. A. L. Friedberg, Professor and Head of the Department of Ceramic Engineering, University of Illinois. 175° taper sections of preheated and enameled 1100 and 5154 were examined.

Fig. #7 is a scanning electron micrograph of the 1100 sample, and Fig. #8 is of 5154. Both samples were etched for 10 seconds with 5% NaOH after cross sectioning and polishing.

In Fig. #8 there appears to be an area where the overlying enamel wedge has chipped and fractured, and in some cases been removed, during the polishing and etching procedure. In back of this zone, where the porcelain enamel becomes continuous, there is a thin, dark layer of material between the porcelain enamel and the aluminum. Whether this layer is of oxidic nature or is a surface layer on the alloy with a composition different from the main body of the alloy is undetermined.

C. Chromium Deposition

The amount of chromium deposited on aluminum during the R-100 chromating pretreatment was studied. This was done by stripping the chromium from the aluminum and analyzing the amount colorimetrically.

The results are listed in Table A. As can be seen, the test results reflect not only the chromium applied in pretreatment, but also to some extent the chromium present in the alloy itself. It is virtually impossible to perform this analysis without extracting some chromium from the body of the alloy. Whether or not the figures have any true significance is thus open to question. We can conclude, however, that most of the chromium being introduced by the pretreatment comes during the alkaline-chromate bath and very little plates out during the R-100 step. The total amount of chromium applied (measured as Cr) is also relatively low.

D. Atomic Absorption Spectrometry

More extensive work was done in this area, after the preliminary results reported on previously. This involved analyzing used spall test solutions for Mg and Cr. The results are included herein as Appendix A. In summary various alloy-pretreatment combinations were tested individually for spall. After testing, the used spall test solutions were examined by atomic absorption spectrometry for Mg and Cr. Those combinations showing the most spall showed the most magnesium in the used test solutions. It is difficult to say whether

this is due to magnesium being extracted from the interface area only or whether it is due to attack of the metal. Prefired samples tended to show higher amounts of magnesium than pickled samples, although not necessarily more spall.

E. Humidity Spall

It has been reported that at times conditions of high humidity would cause spall. This condition would be ideal for examining spalled samples, as no corrosive solution would be present to attack the metal exposed by spalling or to attack any compounds which might be present at the interface side of the spalled enamel flakes.

Prefired and enameled 5086 was exposed at H. H. Robertson Co., Aluminum Company of America and NBS under high humidity conditions. Both H. H. Robertson and Alcoa reported no spalling after exposure times of up to several weeks. The samples exposed at NBS were at constant 100°F and 100% Relative Humidity and showed no spalling after four months. At the end of this time however, the water supply was inadvertently cut off, the humidity dropped to a very low level, and the temperature rose to approximately 275°F. When the exposure conditions were noted to have changed in this manner the samples were checked and found to have spalled severely. It would appear from this, that some fluctuations in temperature and/or humidity are necessary to make spalling occur, or at least to decrease the time necessary for spalling to occur.

F. Electron Microprobe

Three slightly different approaches were used for continuing electron microprobe work.

In the first approach, a chromium free version of the test enamel was applied to prefired and enameled and pickled and enameled samples of 1100, 5053 and 5086. These samples were microprobed for Mg and Cr by Kaiser Aluminum and Chemical Sales, Inc. to attempt to determine if these elements were diffusing or remaining stationary. As has been observed on several previous occasions, Mg was not detected at the interface on the two magnesium bearing alloys, although both exhibited extreme spall failure in both the prefired and pickled conditions. The 1100 samples did not spall. Chromium concentrations were noted at the interfaces of the three pickled samples. This was most likely due to the chromate deposition step, as no chromium concentrations were noted on the prefired and enameled samples. Sodium concentrations were noted at the interface on all 6 samples. No evidence of Cr or Mg diffusion into the porcelain enamel was noted.

A second approach using electron microprobe analysis was on extended fire samples. Specimens of 1100 prefired and enameled, 6061 prefired and enameled and pickled and enameled and 5086 prefired and enameled and pickled and enameled were examined. The special chromium free enamel was used, and the firing time was 10 hours at 1000°F instead of the normal 10 minutes firing time at 1000°F. It was hoped that the longer firing time would broaden the reaction zone between the porcelain enamel and the aluminum and allow a better understanding of relative reaction or diffusion rates of the various components, and particularly Cr, Mg and Al. Reynolds Metals Company is performing this analysis, and their preliminary results indicate that this may be a valuable approach.

The third approach was to examine humidity spalled samples (as mentioned above) by electron microprobe. The Aluminum Company of America performed this analysis. The material examined was spalled 5086 which had been prefired and enameled. They examined unspalled areas, the surface of the metal exposed by the spalling, and the back or interface side of spalled enamel flakes. Their findings indicate again that in unspalled areas, magnesium has a tendency to collect at the interface. In this case, the concentration was not continuous across the interface, but occurred in more or less isolated patches. In areas where spalling had exposed the underlying alloy, Mg was found still attached to the surface of the metal. Mg was also found to be attached to the interface side of the spalled enamel. Thus it appears that at least a minute amount of Mg is being taken into solution. The porcelain enamel is adhering to the Mg or Mg rich layer, and failure is occurring within this layer.

IV. Continuing Work

As the program continues, more information will be sought in the following areas.

The solution of the oxide layer by the porcelain enamel will be studied further. The effects of chromium on the solution of the layer will be studied, in order to ascertain more clearly how the chromium is affecting adherence. The effect of aluminum in the porcelain enamel on the solution rate will be studied also. Aluminum oxide will be introduced into the porcelain enamel before firing, to see whether this markedly affects the solution rate of the oxide layer by the porcelain enamel.

Additional work is planned with the electron microprobe on extended fire samples to further investigate diffusion and interface reactions. A broader range of firing times will be studied, with the firing times varying from 10 minutes to 10 hours. The incidence of spall as a function of firing time will be determined on the same range.

Another attempt at electron microscopy of anodized, polished cross sections will be made. In this series, we will revert to the 5° taper sections, with the aluminum thus forming the thin wedge. This will eliminate the enamel chippage at the interface and hopefully, the anodizing process will not attack too severely and affect removal of the tapered metal back from the interface.

As a result of the behavior of the samples in the humidity spall test, investigations will be made of heat-cool cycling coupled with humidity. Perhaps this approach will develop a useful insight into spall failure.

TABLE A

Amount of Chromium
in milligrams/square foot

Alloy	Condition		
	No Pretreatment	Clean + R-100*	Fully Pretreated (which includes alkali chromate bath)
1100	0.00	0.05	0.80
6061	0.56	0.79	1.76
5086	0.08	0.21	1.54

* R-100 Chemical Pretreatment for Aluminum Pat. #2,719,796 (Aluminum Co. of America). A chromic acid-sulfuric acid bath used to deoxidize or desmut the aluminum alloy surface.

Appendix
A

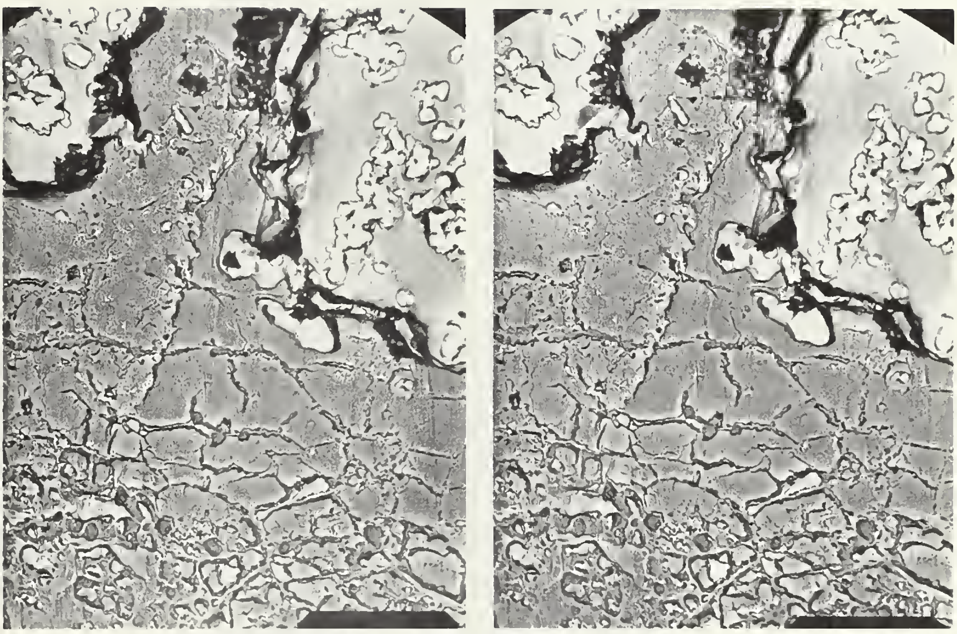

Fig. #1 Prefired and enameled 6061 alloy
175° taper section - etched
5.6 KX Stereo pair

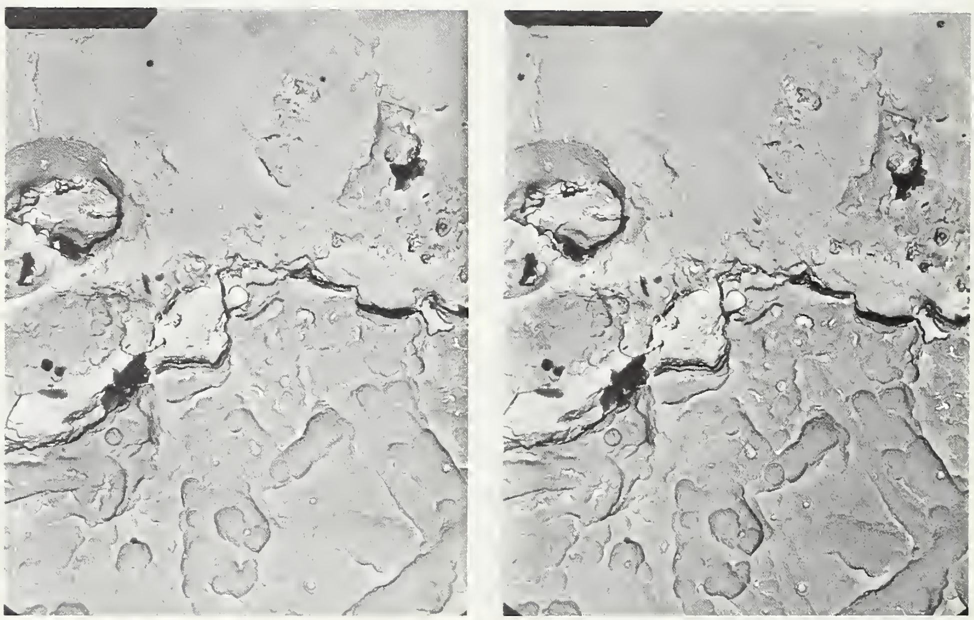

Fig. #2 Prefired and enameled 6061 alloy
175° taper section - etched
5.6 KX Stereo pair

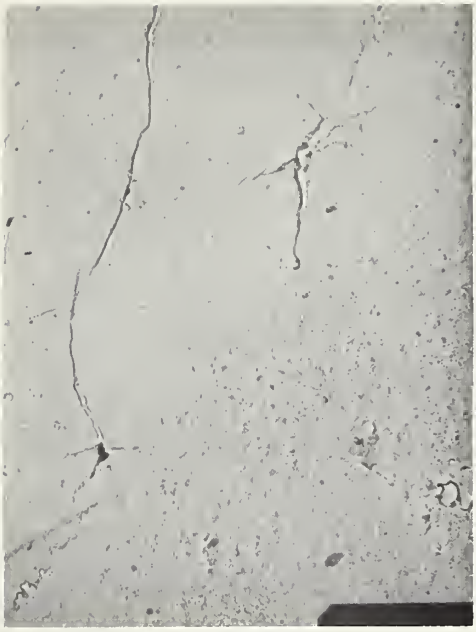

Fig. #3 Prefired and enameled 1100 alloy
175° taper section - anodized after polishing
4 KX

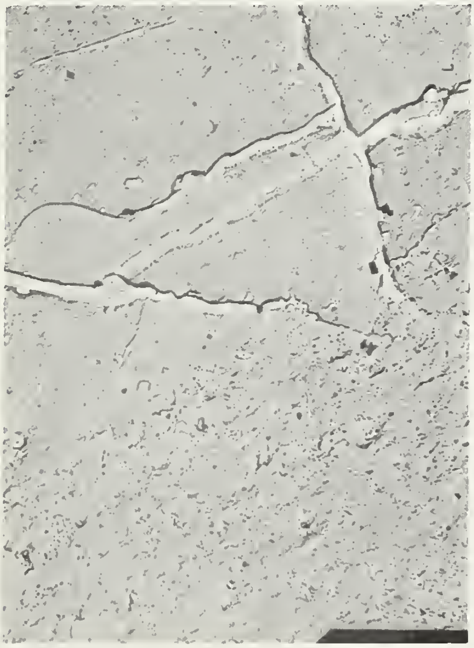

Fig. #4 Prefired and enameled 1100 alloy
175° taper section - anodized after polishing
8 KX

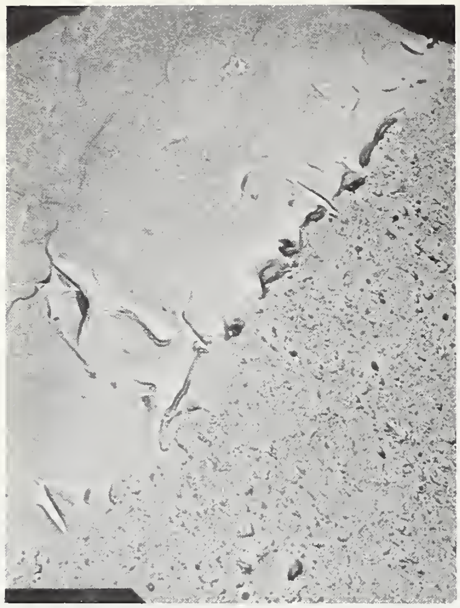

Fig. #5 Prefired and enameled 5154 alloy
175° taper section - anodized after polishing
8 KX

Fig. #6 Prefired and enameled 5154 alloy
175° taper section - anodized after polishing
4 KX

Fig. #7 Prefired and enameled 1100
Scanning Electron Microscope 1,642 X

Fig. #8 Prefired and enameled 5154
Scanning electron microscope 1,642X

The following pages contain the results of analyses made on various spall test solutions after testing various aluminum alloy-pretreatment combinations in them. The analytical methods were refined somewhat, and correction factors were included where they applied. These results thus supersede previously issued data. Mr. M. E. Reed, Kaiser Aluminum & Chemical Corporation, performed the analyses on a Perkin-Elmer, Model 303 Atomic Absorption Spectrophotometer using a recorder and both nitrous oxide and air-acetylene flames.

The results on the next two pages are from 3" x 4" panels enameled on both sides. Before spall testing, the samples were scored at 1/2-inch intervals in both directions on both sides. Two such samples were totally submersed in 700 cc of solution in each beaker. Submersion, or test times, were 20 hours for the 1% $SbCl_3$ solutions and 96 hours for the distilled water and 5% NH_4Cl solutions.

Enameled 3" x 4" Panels

Alloy	Pretreatment	Spall Solution	Total Milligrams of Magnesium	% Bare Metal
1100	prefire	distilled water	0.00	0%
1100	pickled	"	0.00	1%*
3003	prefire	"	0.00	1%*
3003	pickled	"	0.00	1%*
6061 (1)	prefire	"	0.07	1%*
6061	pickled	"	0.06	1%*
6061-X (2)	prefire	"	0.00	0%
6061-X	pickled	"	0.00	1%*
5086	prefire	"	0.03	0%
5086	pickled	"	0.15	1%*
1100	prefire	5% NH_4Cl	0.00	1%*
1100	pickled	"	0.00	1%*
3003	prefire	"	0.00	3%
3003	pickled	"	0.00	2%
6061	prefire	"	0.44	1%*
6061	pickled	"	0.59	4%
6061-X	prefire	"	2.39	2%
6061-X	pickled	"	2.00	1%*
5086	prefire	"	7.76	70%
5086	pickled	"	4.20	20%
1100	prefire	1% $SbCl_3$	0.00	0%
1100	pickled	"	0.06	0%
3003	prefire	"	0.24	85%
3003	pickled	"	0.07	35%
6061	prefire	"	2.55	5%
6061	pickled	"	0.43	60%
6061-X	prefire	"	2.73	30%
6061-X	pickled	"	2.17	5%
5086	prefire	"	17.81	100%
5086	pickled	"	16.51	100%

* This percentage of bare area occurred mostly at marks caused by pins on which the sample rested during firing.

(1) The samples marked 6061 were from the lot of 6061 that appeared to give reverse results on spall testing (i.e.: prefired samples did not spall but pickled samples did).

(2) The samples marked 6061-X were from a lot of 6061 which gave expected spall results (i.e.: prefired samples showed some spalling, but pickled samples did not).

Enameled 3" x 4" Panels

Alloy	Pretreatment	Spall Solution	Total Milligrams of Chromium	% Bare Metal
1100	prefire	distilled water	0.01	0%
1100	pickled	"	0.17	1%*
3001	prefire	"	0.16	1%*
3003	pickled	"	0.14	1%*
6061 (1)	prefire	"	0.14	1%*
6061	pickled	"	0.13	1%*
6061-X (2)	prefire	"	$<$ 0.01	0%
6061-X	pickled	"	$<$0.01	1%*
5086	prefire	"	0.12	0%
5086	pickled	"	0.02	1%*
1100	prefire	5% NH_4Cl	0.08	1%*
1100	pickled	"	0.08	1%*
3003	prefire	"	0.01	3%
3003	pickled	"	0.13	2%
6061	prefire	"	0.05	1%*
6061	pickled	"	0.07	4%
6061-X	prefire	"	0.02	2%
6061-X	pickled	"	0.07	1%*
5086	prefire	"	0.21	70%
5086	pickled	"	0.56	20%
1100	prefire	1% $SbCl_3$	0.16	0%
1100	pickled	"	0.10	0%
3003	prefire	"	0.17	85%
3003	pickled	"	0.17	35%
6061	prefire	"	0.65	5%
6061	pickled	"	1.15	60%
6061-X	prefire	"	0.29	30%
6061-X	pickled	"	0.29	5%
5086	prefire	"	0.33	100%
5086	pickled	"	1.65	100%

* This percentage of bare area occurred mostly at marks caused by pins on which the sample rested during firing.

(1) The samples marked 6061 were from the lot of 6061 that appeared to give reverse results on spall testing (i.e.: prefired samples did not spall, but pickled samples did).

(2) The samples marked 6061-X were from a lot of 6061 which gave expected spall results (i.e.: prefired samples showed some spalling, but pickled samples did not).

The results on the following two pages are from 2" x 3" unenameled panels. Single samples of each alloy-pretreatment combination were submersed in 350 cc of the spall test solutions. The exposure times were 20 hours for the $SbCl_3$ and 96 hours for the distilled water and NH_4Cl solutions.

Unenameled 2" x 3" Panels

Alloy	Pretreatment	Test Solution	Total Millegrams of Magnesium
1100	prefire	distilled water	0.00
1100	pickled	"	0.00
3003	prefire	"	0.00
3003	pickled	"	0.01
6061	prefire	"	0.36
6061	pickled	"	0.00
6061-X	prefire	"	0.47
6061-X	pickled	"	0.00
5086	prefire	"	0.97
5086	pickled	"	0.05
1100	prefire	5% NH_4Cl	0.00
1100	pickled	"	0.00
3003	prefire	"	0.00
3003	pickled	"	0.00
6061	prefire	"	0.39
6061	pickled	"	0.04
6061-X	prefire	"	1.26
6061-X	pickled	"	0.58
5086	prefire	"	0.97
5086	pickled	"	0.12
1100	prefire	1% $SbCl_3$	0.00
1100	pickled	"	0.00
3003	prefire	"	0.02
3003	pickled	"	0.01
6061	prefire	"	0.90
6061	pickled	"	1.27
6061-X	prefire	"	2.73
6061-X	pickled	"	2.17
5086	prefire	"	4.56
5086	pickled	"	5.45

Unenameled 2" x 3" Panels

Alloy	Pretreatment	Test Solution	Total Milligrams of Chromium
1100	prefire	distilled water	<0.01
1100	pickled	"	0.02
3003	prefire	"	0.02
3003	pickled	"	0.03
6061	prefire	"	0.02
6061	pickled	"	0.03
6061-X	prefire	"	<0.01
6061-X	pickled	"	<0.01
5086	prefire	".	0.03
5086	pickled	"	0.01
1100	prefire	5% NH_4Cl	0.07
1100	pickled	"	0.01
3003	prefire	"	<0.01
3003	pickled	"	0.04
6061	prefire	"	0.02
6061	pickled	"	0.02
6061-X	prefire	"	0.03
6061-X	pickled	"	0.05
5086	prefire	"	0.06
5086	pickled	"	0.02
1100	prefire	1% $SbCl_3$	0.04
1100	pickled	"	0.35
3003	prefire	"	0.07
3003	pickled	"	0.65
6061	prefire	"	0.15
6061	pickled	"	1.05
6061-X	prefire	"	0.17
6061-X	pickled	"	0.32
5086	prefire	"	0.14
5086	pickled	"	0.58

An additional group of tests was run in order to try to determine the cause of magnesium release into solution. For this test 5086 was used.

Test #1

Pickled and prefired samples were exposed for 20 hours to the liquid phase of the mill addition. This includes Boric Acid, KOH, Potassium Silicate and water. After exposing 1 2" x 3" panel of each pretreatment condition to the mill addition, this solution was analyzed. A blank consisting of unused liquid mill addition was also run. The results below reflect correction for Mg and Cr found in the blank. 300 cc of solution was used.

	Mg (milligrams)	Cr (milligrams)
preheat 5086	0.50	0.02
pickled 5086	0.49	0.01

Test #2 (Wet Firing)

In this test, pickled and prefired samples were wet with the liquid phase of the mill addition and immediately fired at 1000°F for 10 minutes. After firing the samples were then submerged in 300 cc of 1%SbCl for 20 hours. Following exposure of the test samples, the test solutions were then analyzed.

	Mg (milligrams)	Cr (milligrams)
preheat 5086	5.82	0.13
pickled 5086	4.56	0.25

Test #3 (Dry Firing)

In this test, the samples were dried after exposure to the liquid mill addition phase. When dry, the samples were fired, exposed for 20 hours in 300 cc 1% $SbCl_3$ and then the test solutions were analyzed.

	Mg (milligrams)	Cr (milligrams)
preheat 5086	5.68	0.13
pickled 5086	5.37	0.29

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